Structural and Mechanistic Investigations of the Oxidation of Dimethylplatinum(II) Complexes by Dioxygen

Vsevolod V. Rostovtsev, Larry Henling, Jay A. Labinger* and

John E. Bercaw*

Arnold and Mabel Beckman Laboratories of Chemical Synthesis

California Institute of Technology, Pasadena, California 91125 (U.S.A.)

Supporting Information: Details of the structure determinations of (tmeda)Pt(OH)(OCH_3)(CH_3)$_2$ (**3**) and (tmeda)Pt(OOH)(OCH_3)(CH_3)$_2$ (**2**), including atomic coordinates, bond lengths and angles.

Structure Determination of (tmeda)Pt(OH)(OCH_3)(CH_3)$_2$ (3).

Figure S1. Labeled Diamond view of **3**, with 50% ellipsoids. Hydrogen atoms other than H0A have been omitted.

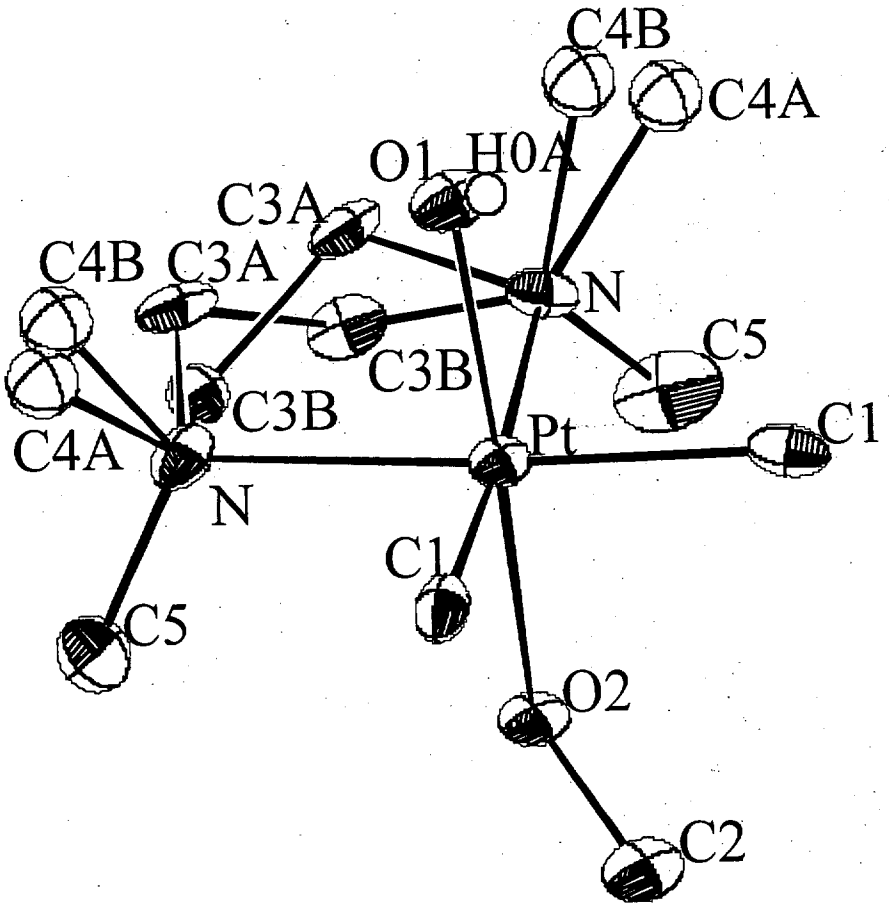

Figure S2. Crystal packing in **3** viewed along the *b* axis, showing the unit cell boundaries, with 50% ellipsoids. Hydrogen atoms have been omitted. Note intermolecular hydrogen bonds.

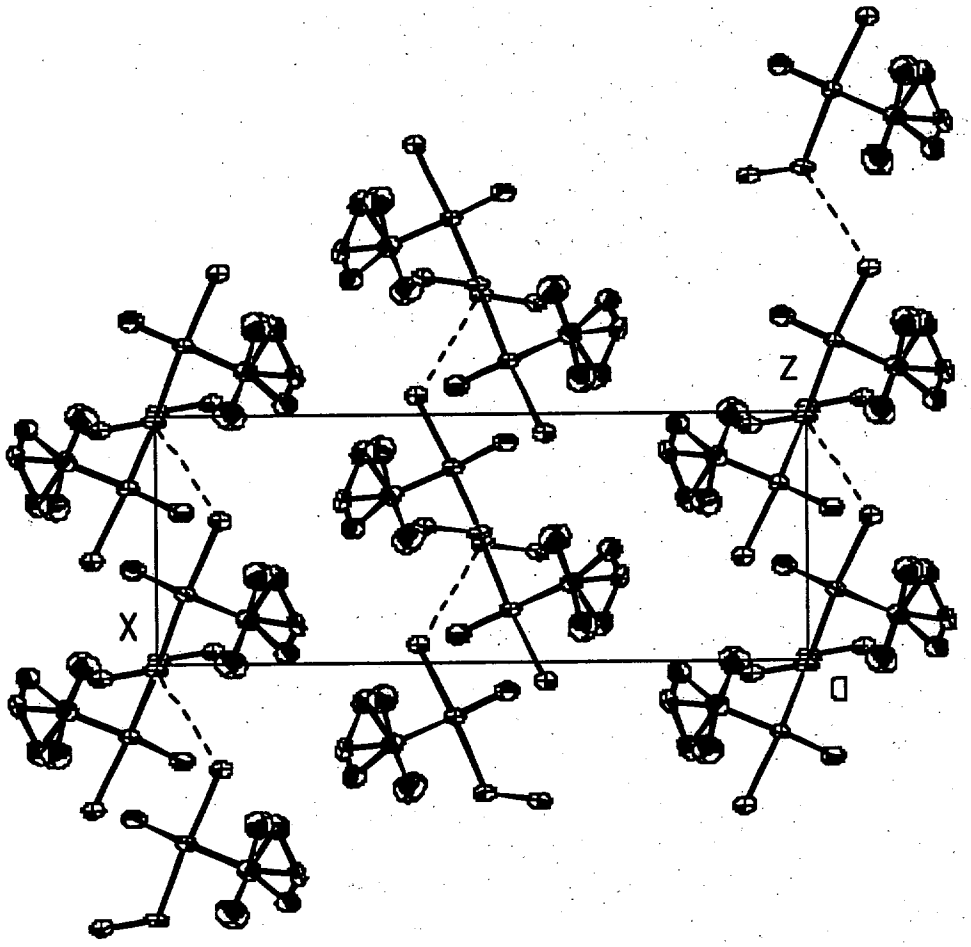

Table S1. Crystal Structure Determination Details for **3**.

Crystal Data Details for **3**		
Empirical formula	$C_9H_{26}N_2O_2Pt$	
Formula weight	389.41	
Crystallization solvent	methanol	
Crystal shape	prismatic	
Crystal color	colorless	
Crystal size	0.11 x 0.13 x 0.15 mm	
Data Collection		
Type of diffractometer	CAD-4	
Wavelength	0.71073 Å MoKα	
Data collection temperature	84 K	
Theta range for 25 reflections used in lattice determination	13 to 14°	
Unit cell dimensions	a = 16.101(5) Å	α= 90°
	b = 13.245(4) Å	β= 90°
	c = 6.134(2) Å	γ = 90°
Volume	1308.1(7) $Å^3$	
Z	4	
Crystal system	orthorhombic	
Space group	Pnma (# 62)	
Density (calculated)	1.977 g/cm^3	
F(000)	752	
Theta range for data collection	1.5 to 25°	
Completeness to theta = 25°	100.0%	
Index ranges	$-19 \le h \ge 19, -15 \le k \ge 0, -7 \le l \ge 7$	
Data collection scan type	ω-scan	
Reflections collected	5701	
Independent reflections	1203 [R_{int}= 0.013]	
Reflections > 2s(I)	1026	
Average s(I)/(net I)	0.0299	
Absorption coefficient	10.71 mm^{-1}	
Absorption correction	ψ-scan (North, 1968)	
Max. and min. transmission	1.10 and 0.89	

Reflections monitored for decay	75
Decay of standards	0%

Structure Solution and Refinement

Primary solution method	direct methods
Secondary solution method	difference map
Hydrogen placement	calculated
Refinement method	full-matrix least-squares on F^2
Data / restraints / parameters	1203 / 0 / 133
Treatment of hydrogen atoms	coordinates refined, U_{iso} fixed at 120% U_{eq} of attached atom
Goodness-of-fit on F^2	1.828
Final R indices	
[I>2s(I), 1026 reflections]	R1 = 0.0297, wR2 = 0.0533
R indices (all data)	R1 = 0.0460, wR2 = 0.0593
Type of weighting scheme used	sigma
Weighting scheme used	$\omega=1/\sigma^2(Fo^2)$
Max shift/error	0.155
Average shift/error	0.011
Largest diff. peak and hole	1.62 and -2.31 e·Å^{-3}

Programs Used

Cell refinement	CAD-4 Software (Enraf-Nonius, 1989)
Data collection	CAD-4 Software (Enraf-Nonius, 1989)
Data reduction	CRYM (Duchamp, 1964)
Structure solution	SHELXS-97 (Sheldrick, 1990)
Structure refinement	SHELXL-97 (Sheldrick, 1997)
Graphics	Diamond, Bruker SHELXTL v5.1

References

Diamond 2.1. (2000) Crystal Impact GbR, Bonn, Germany.

Duchamp, D. J. (1964). Am. Crystallogr. Assoc. Meet., Bozeman, Montana., Paper B14, 29-30

Nonius. (1989). CAD-4 Software. Version 5. Nonius, Delft, The Netherlands
Spek, A.L. (1990). *Acta Cryst.*, A46, C-34.
Sheldrick, G. M. (1990). *Acta Cryst.*, A46, 467-473.
Sheldrick, G. M. (1997). SHELXL-97. Program for Structures Refinement. Univ. of Gottingen, Federal Republic of Germany

Special Refinement Details

Four crystals were examined on the diffractometer; data were only collected for the last. The first three crystals were discarded due to broad diffraction peaks and severe indexing problems. The crystals were mounted on a glass fiber with Paratone-N oil. Data were collected with 1.5° ω-scans because of the poor crystal quality.

The individual backgrounds were replaced by a background function of two theta derived from weak reflections with $I < 3\sigma(I)$. For point group *mmm*, the GOF_{merge} was 1.08 (1203 multiples); R_{int} was 0.013 for 94 duplicates (there are only a few duplicates since four equivalent sets of reflections were collected). __scan data were used for the absorption correction. There was no decay. Weights were calculated as $1/\sigma^2(F_o^2)$; variances ($\sigma^2(F_o^2)$) were derived from counting statistics plus an additional term, $(0.014I)^2$; variances of the merged data were obtained by propagation of error plus another additional term, $(0.014\langle I\rangle)^2$.

No reflections were specifically omitted from the final processed dataset. Refinement of F^2 was against all reflections. The weighted R-factor (ωR) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt), and is not relevant to the choice of reflections for refinement.

The molecule lies on a mirror plane, which contains the heavy atoms Pt, O1, O2, C2 and bisects the tmeda. The tmeda backbone carbon C3 and one of the methyl groups, C4, are each disordered over two sites (C3A, C3B and C4A, C4B) with occupancy 1/2 since C3A is bonded to the mirror image of C3B. The coordinates of all hydrogen atoms including the half-populated sites were refined with U_{iso}'s fixed at 120% of the U_{eq} of the attached atom. All bonds involving the hydrogen atoms are reasonable given the large esd's. There is a hydrogen bond between O1 and the O2 at x, y, z-1 (O1···O2 is 2.946(9)Å and the

O1-H1A-O2 angle is 172(18)°), which forms a linear chain of molecules along the *c* axis.

The largest shift/esd of 0.155 is for the x coordinate of the half-hydrogen H3AA which oscillates approximately 0.05Å from cycle to cycle. Seventeen peaks in the final difference map have intensities ≥ |1| $e \cdot Å^{-3}$; the two largest are -2.31 $e \cdot Å^{-3}$ (0.92Å from Pt) and 1.62 $e \cdot Å^{-3}$ (0.89Å from Pt). Nine of these peaks, with intensities up to 1.45 $e \cdot Å^{-3}$ and –1.53 $e \cdot Å^{-3}$, are in the vicinity of the disordered region. There are several close intermolecular contacts between both H0A and H5A (C5 was not split into two sites) and the hydrogens in the disordered region. Clearly, there are some subtleties to the disorder/packing that are not readily modeled.

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3.** U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Pt	4569(1)	7500	2137(1)	24(1)
O1	4018(5)	7500	-776(11)	38(2)
O2	5027(5)	7500	5218(10)	29(2)
N	3613(3)	6391(5)	3215(8)	31(1)
C1	5369(5)	6432(6)	1148(11)	32(2)
C2	5864(7)	7500	5534(17)	37(3)
C3A[a]	2863(12)	6983(16)	3420(30)	36(5)
C4A[a]	3439(15)	5580(20)	1410(30)	40(5)[b]
C3B[a]	3022(12)	7046(14)	4540(40)	34(5)
C4B[a]	3169(15)	5886(19)	1500(30)	41(5)[b]
C5	3875(7)	5729(9)	4983(17)	60(3)

[a] Population: 1/2

[b] U_{iso}

Table S3. Bond lengths [Å] and angles [°] for **3**.

Pt-O1	1.995(7)
Pt-C1	2.007(8)
Pt-O2	2.028(6)
Pt-N	2.229(6)
O1-H0A	0.55(12)
O2-C2	1.362(13)
N-C4B	1.436(19)
N-C3A	1.445(19)
N-C5	1.457(11)
N-C3B	1.52(2)
N-C4A	1.57(2)
C1-H1A	1.00(7)
C1-H1B	0.91(8)
C1-H1C	0.89(8)
C2-H2A	1.00(11)
C2-H2B	0.94(7)
C3A-C3B^{i}	1.48(2)
C3A-H3AA	1.0(3)
C3A-H3AB	0.91(14)
C4A-H4AA	1.13(16)
C4A-H4AB	0.94(17)
C4A-H4AC	1.0(2)
C3B-H3BA	0.9(3)
C3B-H3BB	1.09(13)
C4B-H4BA	1.19(16)
C4B-H4BB	1.1(2)
C4B-H4BC	0.86(18)
C5-H5A	1.08(10)
C5-H5B	0.83(10)
C5-H5C	0.95(10)
C4A···C4B	0.60(3)
C3A···C3B	0.74(2)
O1-Pt-C1	90.8(3)
C1^{i}-Pt-C1	89.6(5)
O1-Pt-O2	174.9(3)
C1-Pt-O2	92.8(3)
O1-Pt-N	87.6(2)
C1^{i}-Pt-N	176.1(3)
C1-Pt-N	93.9(3)
O2-Pt-N	88.6(2)
N-Pt-N^{i}	82.5(3)
Pt-O1-H0A	113(10)
C2-O2-Pt	119.5(6)
C4B-N-C5	114.2(11)
C3A-N-C5	120.3(10)
C4B-N-C3B	110.1(14)
C5-N-C3B	97.3(10)
C3A-N-C4A	106.5(14)
C5-N-C4A	99.4(12)
C4B-N-Pt	115.7(9)

C3A-N-Pt	104.2(9)
C5-N-Pt	114.6(5)
C3B-N-Pt	102.4(8)
C4A-N-Pt	111.5(8)
Pt-C1-H1A	113(4)
Pt-C1-H1B	112(5)
H1A-C1-H1B	111(6)
Pt-C1-H1C	112(5)
H1A-C1-H1C	112(6)
H1B-C1-H1C	96(6)
O2-C2-H2A	115(6)
O2-C2-H2B	116(5)
H2A-C2-H2B	100(6)
N-C3A-H3AA	120(10)
N-C3A-H3AB	123(9)
H3AA-C3A-H3AB	95(10)
N-C4A-H4AA	108(8)
N-C4A-H4AB	103(10)
H4AA-C4A-H4AB	94(10)
N-C4A-H4AC	94(10)
H4AA-C4A-H4AC	126(10)
H4AB-C4A-H4AC	130(10)
N-C3B-H3BA	106(10)
N-C3B-H3BB	111(7)
H3BA-C3B-H3BB	104(10)
N-C4B-H4BA	111(8)
N-C4B-H4BB	93(10)
N-C4B-H4BC	102(10)
H4BA-C4B-H4BC	102(10)
H4BB-C4B-H4BC	120(10)
N-C5-H5A	111(5)
N-C5-H5B	116(7)
H5A-C5-H5B	106(8)
N-C5-H5C	110(6)
H5A-C5-H5C	108(7)
H5B-C5-H5C	104(9)

Structure Determination of (tmeda)Pt(OOH)(OCH_3)(CH_3)$_2$ (2).

Figure S3. Unlabeled Diamond view of **2**, with 50% ellipsoids (only Pt is anisotropic). Orientation A is in black and orientation B in dark grey. Hydrogen atoms are at arbitrary scale.

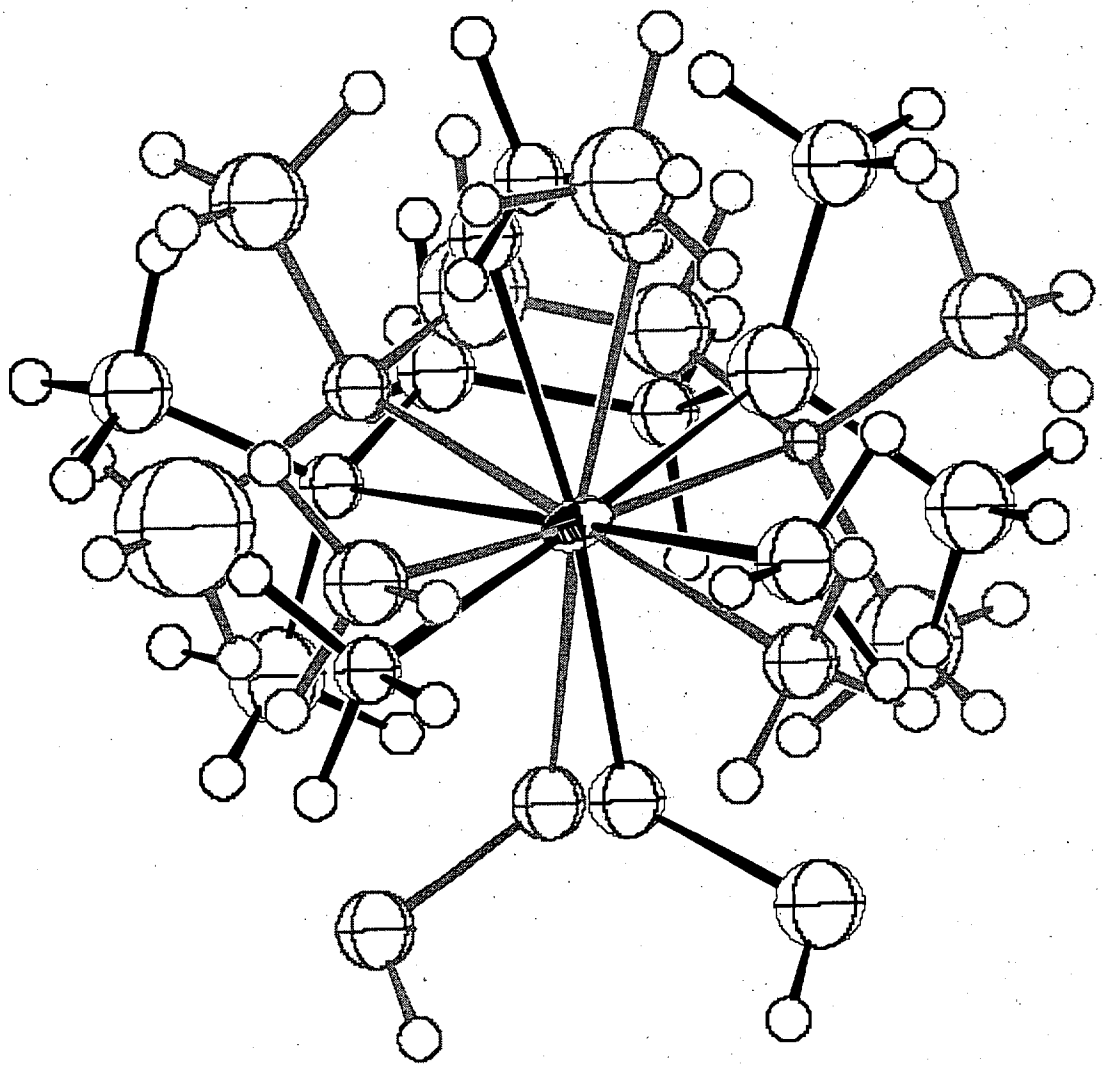

Figure S4. Unlabeled Diamond view of an A(x+1,y,z; top), B(x,y,z; bottom) pair of molecules of **2** showing the hydroperoxy-methoxy hydrogen bonding.

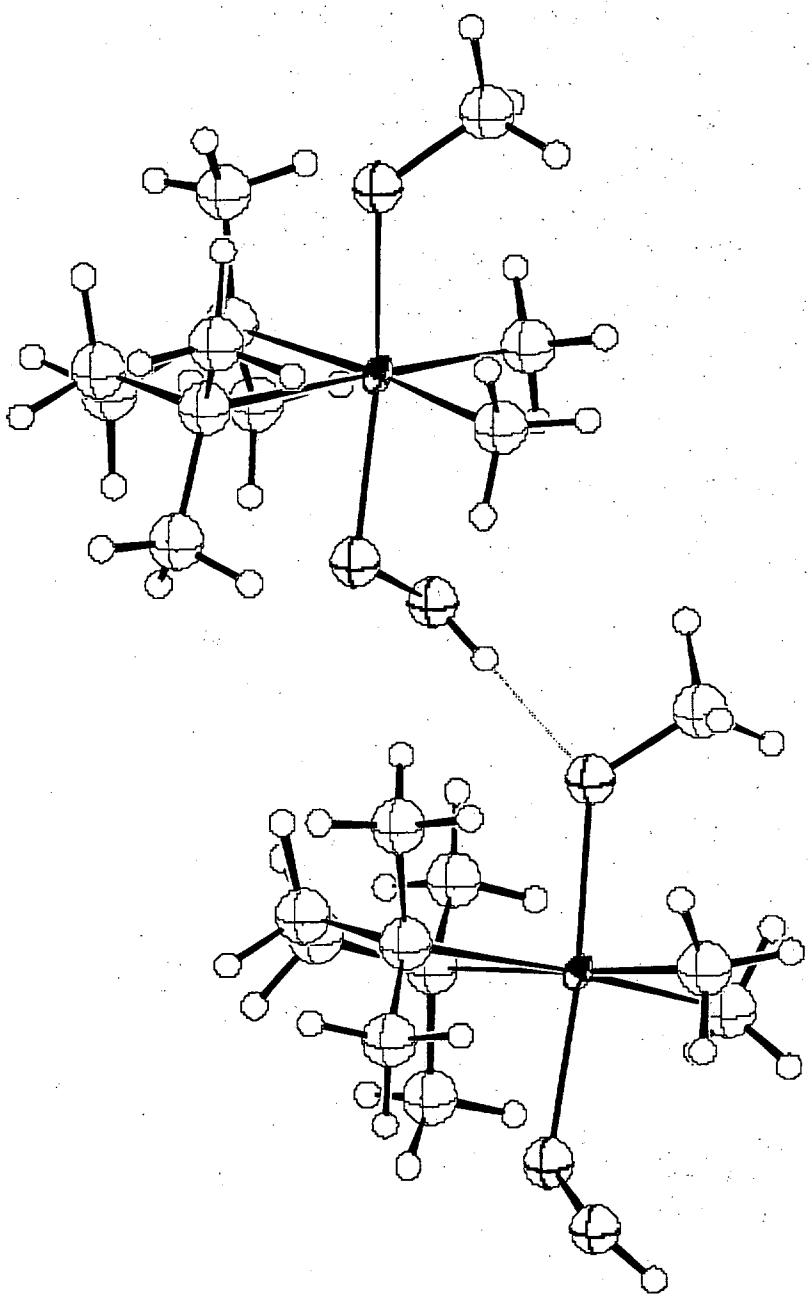

Table S4. Crystal Structure Determination Details for **2**.

Crystal Data Details for **2**		
Empirical formula	$C_9H_{26}N_2O_3Pt$	
Formula weight	405.41	
Crystallization solvent	acetone	
Crystal shape	irregular thin wedge	
Crystal color	colorless	
Crystal size	0.07 x 0.21 x 0.22 mm	
Data Collection		
Preliminary photograph(s)	rotation	
Type of diffractometer	Bruker SMART 1000 ccd	
Wavelength	0.71073 Å MoKα	
Data collection temperature	98 K	
Theta range for 8290 reflections used in lattice determination	2.7 to 28.4°	
Unit cell dimensions	a = 6.5985(8) Å	α= 90°
	b = 13.5019(16) Å	β= 93.408(2)°
	c = 14.9187(18) Å	γ = 90°
Volume	1326.8(3) $Å^3$	
Z	4	
Crystal system	monoclinic	
Space group	P $2_1/n$ (# 14)	
Density (calculated)	2.030 g/cm^3	
F(000)	784	
Theta range for data collection	2.0 to 25.0°	
Completeness to theta = 25.00°	100.0%	
Index ranges	$-7 \le h \ge 7, -16 \le k \ge 16, -17 \le l \ge 17$	
Data collection scan type	ω scans at 5 fixed ϕ values	
Reflections collected	16295	
Independent reflections	2335 [R_{int}= 0.0741]	
Reflections > 2s(I)	2096	
Average s(I)/(net I)	0.0424	
Absorption coefficient	10.57 mm^{-1}	
Absorption correction	empirical	
Max. and min. transmission	1.000 and 0.471	

Reflections monitored for decay	first 75 scans recollected at end of runs
Decay of standards	0%

Structure Solution and Refinement

Primary solution method	direct methods
Secondary solution method	difference map
Hydrogen placement	calculated
Refinement method	full-matrix least-squares on F^2
Data / restraints / parameters	2335 / 0 / 123
Treatment of hydrogen atoms	not refined, U_{iso} fixed at 120% U_{eq} of attached atom
Goodness-of-fit on F^2	2.20
Final R indices	
[I>2s(I), 2096 reflections]	R1 = 0.0458, wR2 = 0.0990
R indices (all data)	R1 = 0.0516, wR2 = 0.1001
Type of weighting scheme used	sigma
Weighting scheme used	$\omega=1/\sigma^2(Fo^2)$
Max shift/error	0.020
Average shift/error	0.001
Largest diff. peak and hole	2.98 and -2.73 e·Å^{-3}

Programs Used

Cell refinement	Bruker SMART v5.606, Bruker SAINT v6.02
Data collection	Bruker SMART v5.060
Data reduction	Bruker SAINT v6.02
Structure solution	SHELX-97 (Sheldrick, 1997)
Structure refinement	SHELX-97 (Sheldrick, 1997)
Graphics	Diamond, Bruker SHELXTL v5.1

References

Bruker (1999) SMART (v5.060), SMART (v5.606), SAINT (v6.02) and SHELXTL (v5.1). Bruker AXS Inc., Madison, Wisconsin, USA.

Diamond 2.1. (2000) Crystal Impact GbR, Bonn, Germany.

Spek, A.L. (1990). *Acta Cryst.*, A**46**, C-34.

Sheldrick, G. M. (1997). SHELXL-97. Program for Structures Refinement. Univ. of Gottingen, Federal Republic of Germany.

Special Refinement Details

A semi-opaque, rounded thin wedge was selected from a batch of freshly grown crystals and mounted on a glass fiber with Paratone-N oil. This sample was the best of many chosen from a number of crystallizations; data collected at room temperature were no better than low-temperature data. Five runs of data were collected with 15 second long, –0.3° wide ω-scans at five values of φ(0, 120, 240, 180, and 300°) with the detector 5 cm (nominal) distant at a θ of -28°. The spots were broad and sometimes split. The initial cell for data reduction was calculated from 999 centered reflections chosen from throughout the data frames. A total of 41 reflections was discarded in the triclinic least-squares with a reciprocal lattice vector tolerance of 0.008 (0.005 is customary). Of the rejects, 20 had substantially non-integral indices and presumably are from a small fragment with a different orientation. There was no evidence of a larger cell. For data processing with SAINT v6.02, all defaults were used, except: a fixed box size of 2.0 x 2.0 x 1.2 was used, periodic orientation matrix updating was disabled, the instrument error was set to zero, no Laue class integration restraints were used, and for the post-integration global least squares refinement, no constraints were applied. The profiles exhibited an apparently considerable truncation of many reflections, especially in the z-direction. A number of both larger and smaller box sizes were tried but gave worse refinements of the structure. Both the initial cell refinement and the global cell refinement gave an ω zero ~ 0.4° too negative. No decay correction was needed. The crystal boundaries were rudely indexed as faces; these were not good enough for a face-indexed absorption correction. A SADABS v2.03 beta correction was applied (relative correction factors: 1.000 to 0.471) with g = 0.113, 100 refinement cycles, and defaults for the remaining parameters. Again, a number of other options such as discarding portions of the data and using different orders of Bessel functions did not lead to a better model refinement.

No reflections were specifically omitted from the final processed dataset but the data were truncated at a 2θ of 50° as the high angle spots seemed fuzzier; 3469 reflections were rejected, with 20 space group-absence violations, 0 inconsistent equivalents, and no reflections suppressed. Refinement of F^2 was against all reflections. The weighted R-factor (ωR) and goodness of fit (Σ) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt), and is not relevant to the choice of reflections for refinement.

The asymmetric unit consists of one molecule of $Pt(tmeda)(CH_3)_2(OOH)(OCH_3)$, which is disordered ~1:1 over two orientations. All atoms except the platinum were split and modeled with two sites. The major difference between the two orientations is the position of the terminal hydroperoxy oxygen atom; it can lie under either of the two methyl groups. The rest of the molecule tilts to accommodate these two positions, as can be seen above. Orientation A has a slightly more reasonable structure than orientation B, which has a carbon atom (C1B of the tmeda backbone) with distorted geometry. This distortion is most likely due to additional disorder of the tmeda, a failing to which this ligand is all too prone; see, e.g., structure 1, $Pt(tmeda)(CH_3)_2(OH)(OCH_3)$. Atom N2B has a small U_{iso} and tmeda methyl carbon C4B has a large U_{iso}. The distances and angles seem reasonable, given the large e.s.d's.

The location of the platinum atom could easily be determined by direct methods or from a Patterson map. The remaining non-hydrogen atoms were laboriously determined from successive structure factor - Fourier calculations. [Early refinement tended to hamper the subsequent location of missing atoms.] When several of the ligand atoms were initially refined anisotropically with one fully occupied site, SHELXL did not suggest splitting them. However, these individual sites were manually divided into two and refined successfully with isotropic displacement parameters. The separation between the two positions for the nine pairs of close atoms (see Table 4) range from 0.50(2) Å to 1.10(1) Å. The platinum atom was refined anisotropically, all other non-hydrogen atoms isotropically. All hydrogen atoms were placed at calculated positions with U_{iso}'s fixed at 120% of the U_{iso}'s of the attached atoms. No restraints or constraints were used. Since the molecule is small and disordered, some chemical assumptions were obviously used in the interpretation of this structure. As a test, both the methoxy carbon and the terminal hydroperoxy oxygen were independently refined as a C/O mixture. The results were a somewhat disappointing ~60% of the appropriate atom. Of the 20 largest peaks in the final difference map, 6 are greater than |2| $e \cdot Å^{-3}$ and another 12 are greater than |1| $e \cdot Å^{-3}$. Seven of these 18 peaks are near the platinum atom, including the largest positive peak of 2.98 $e \cdot Å^{-3}$ at a distance of 0.89 Å. The largest negative peak of –2.73 $e \cdot Å^{-3}$ is 0.75 Å from N2B (anomalously small U_{iso}); the other big excursions not near the platinum are 2.29 $e \cdot Å^{-3}$ at 0.88 Å from C8B, 1.79 $e \cdot Å^{-3}$ at 0.67 Å from C6B, -1.66 $e \cdot Å^{-3}$ at 1.45 Å from O2B, 1.63 $e \cdot Å^{-3}$ at 0.68 Å from N2B,

-1.23 e·Å^{-3} at 0.99 Å from H8BC, -1.21 e·Å^{-3} at 1.07 Å from O2A, 1.13 e·Å^{-3} at 0.76 Å from C4B, and 1.06 e·Å^{-3} at 0.80 Å from O1B. The 6 most disagreeable reflections all have k = 5 as well as F_{calc} weak and less than F_{obs}. This suggests twinning. The 20 reflections discarded in the initial unit cell determination could not be indexed by themselves. No other effort was made to investigate this fairly minor problem.

A Platon analysis of intermolecular contacts suggests two potential hydrogen bonds, each between a donor hydroperoxy OH and an acceptor methoxy O. One bond is between two molecules of orientation A translated one unit along the *a*-axis; the other between two molecules of orientation B similarly related.

D-H	d(D-H)	d(H..A)	<DHA	d(D..A)	A
O2A-H0A	0.840	2.344	151.94	3.111	O3A_a [x+1, y, z]
O2B-H0B	0.840	2.449	151.66	3.213	O3B_b [x+1, y, z]

However, the intermolecular distances also reveal some impossibly short contacts, such as 2.08 Å between O2A (*x,y,z*) and C5A (*x+1,y,z*). Therefore, two molecules of orientation A cannot be adjacent along the *a*-axis and thus molecules of orientation A must comprise no more than 50% of the total molecules in the crystal. The population parameter for orientation A refined to 0.508(9), or essentially 1:1. Thus orientations A and B must alternate along the *a*-axis. This alternation must be occasionally broken since no evidence of a larger cell is seen. There are no appreciable intermolecular platinum – hydroperoxy or hydroperoxy – hydroperoxy interactions. As shown in the hydrogen bond table, there is a good hydrogen bond between the hydroperoxy group of one orientation and the methoxy oxygen of the other orientation.

Table S5. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2**. U(eq) is defined as one-third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq} or U_{iso}
Pt	230.8(0.5)	3025.1(0.2)	1570.9(0.2)	16.2(0.2)*
O1A[a]	2970(20)	2404(12)	1818(11)	25(4)
O2A[a]	3980(20)	2151(9)	985(9)	34(4)
O3A[a]	-2603(19)	3652(9)	1546(8)	23(3)
N1A[a]	-70(30)	2781(11)	3066(11)	18(4)
N2A[a]	-1130(40)	1560(16)	1475(13)	36(6)
C1A[a]	-1100(40)	1842(15)	3110(15)	26(5)
C2A[a]	-670(40)	1084(14)	2410(12)	19(5)
C3A[a]	-1040(30)	3635(15)	3524(13)	30(5)
C4A[a]	1800(40)	2569(17)	3563(15)	39(6)
C5A[a]	-3150(30)	1518(15)	1104(13)	31(5)
C6A[a]	230(30)	896(16)	916(14)	35(5)
C7A[a]	-3240(30)	4289(14)	846(13)	21(5)
C8A[a]	1540(30)	4401(14)	1805(13)	19(5)
C9A[a]	530(40)	3224(15)	251(14)	30(5)
O1B[b]	2990(20)	2621(12)	2092(11)	23(4)
O2B[b]	4180(20)	3387(9)	2543(8)	26(4)
O3B[b]	-2630(20)	3196(9)	1009(8)	18(3)
N1B[b]	-1020(30)	2787(11)	2888(11)	19(4)
N2B[b]	-420(30)	1369(12)	1462(10)	7(4)
C1B[b]	-1890(50)	1810(20)	2900(20)	53(9)
C2B[b]	-1470(40)	1196(18)	2301(16)	36(7)
C3B[b]	-2860(40)	3276(16)	3024(15)	39(6)
C4B[b]	330(60)	3120(30)	3640(20)	83(11)
C5B[b]	-1640(30)	1035(15)	683(13)	30(5)
C6B[b]	1550(40)	790(19)	1372(17)	52(7)
C7B[b]	-3200(50)	4030(20)	509(18)	49(7)
C8B[b]	690(40)	4516(17)	1706(15)	30(6)
C9B[b]	1520(40)	3096(15)	354(13)	23(5)

[a] Population: 0.508(9)
[b] Population: 0.492(9)

* U_{eq}

Table S6. Bond lengths [Å] and angles [°] for **2**.

Pt-O1A	2.006(16)
Pt-O3A	2.051(13)
Pt-C8A	2.069(18)
Pt-C9A	2.01(2)
Pt-N1A	2.276(16)
Pt-N2A	2.17(2)
Pt-O1B	2.015(16)
Pt-O3B	2.033(13)
Pt-C8B	2.04(2)
Pt-C9B	2.05(2)
Pt-N1B	2.200(16)
Pt-N2B	2.281(16)
O1A-O2A	1.48(2)
O2A-H0A	0.8400
O3A-C7A	1.40(2)
N1A-C4A	1.43(3)
N1A-C1A	1.44(3)
N1A-C3A	1.50(3)
N2A-C5A	1.41(3)
N2A-C6A	1.55(3)
N2A-C2A	1.55(3)
C1A-C2A	1.50(3)
C1A-H1AA	0.9900
C1A-H1AB	0.9900
C2A-H2AA	0.9900
C2A-H2AB	0.9900
C3A-H3AA	0.9800
C3A-H3AB	0.9800
C3A-H3AC	0.9800
C4A-H4AA	0.9800
C4A-H4AB	0.9800
C4A-H4AC	0.9800
C5A-H5AA	0.9800
C5A-H5AB	0.9800
C5A-H5AC	0.9800
C6A-H6AA	0.9800
C6A-H6AB	0.9800
C6A-H6AC	0.9800
C7A-H7AA	0.9800
C7A-H7AB	0.9800
C7A-H7AC	0.9800
C8A-H8AA	0.9800
C8A-H8AB	0.9800
C8A-H8AC	0.9800
C9A-H9AA	0.9800
C9A-H9AB	0.9800
C9A-H9AC	0.9800
O1B-O2B	1.44(2)
O2B-H0B	0.8400
O3B-C7B	1.39(3)
N1B-C3B	1.41(3)

N1B-C1B	1.43(3)
N1B-C4B	1.46(4)
N2B-C5B	1.44(2)
N2B-C2B	1.48(3)
N2B-C6B	1.53(3)
C1B-C2B	1.26(3)
C1B-H1BA	0.9900
C1B-H1BB	0.9900
C2B-H2BA	0.9900
C2B-H2BB	0.9900
C3B-H3BA	0.9800
C3B-H3BB	0.9800
C3B-H3BC	0.9800
C4B-H4BA	0.9800
C4B-H4BB	0.9800
C4B-H4BC	0.9800
C5B-H5BA	0.9800
C5B-H5BB	0.9800
C5B-H5BC	0.9800
C6B-H6BA	0.9800
C6B-H6BB	0.9800
C6B-H6BC	0.9800
C7B-H7BA	0.9800
C7B-H7BB	0.9800
C7B-H7BC	0.9800
C8B-H8BA	0.9800
C8B-H8BB	0.9800
C8B-H8BC	0.9800
C9B-H9BA	0.9800
C9B-H9BB	0.9800
C9B-H9BC	0.9800
O1A···O1B	0.50(2)
O3A···O3B	1.01(1)
N1A···N1B	0.67(2)
N2A···N2B	0.54(2)
C1A···C1B	0.59(4)
C2A···C2B	0.57(3)
C7A···C7B	0.61(3)
C8A···C8B	0.59(3)
C9A···C9B	0.69(3)

O1A-Pt-C9A	95.6(9)
O1A-Pt-O3A	170.5(6)
C9A-Pt-O3A	93.9(8)
O1A-Pt-C8A	89.0(8)
C9A-Pt-C8A	89.0(8)
O3A-Pt-C8A	90.1(7)
O1A-Pt-N2A	89.8(7)
C9A-Pt-N2A	97.0(8)
O3A-Pt-N2A	90.0(7)
C8A-Pt-N2A	174.1(7)
O1A-Pt-N1A	83.7(6)
C9A-Pt-N1A	179.2(8)
O3A-Pt-N1A	86.8(6)

C8A-Pt-N1A	91.4(7)
N2A-Pt-N1A	82.7(7)
O1B-Pt-O3B	170.8(5)
O1B-Pt-C8B	96.0(8)
O3B-Pt-C8B	93.2(8)
O1B-Pt-N2B	85.7(6)
O3B-Pt-N2B	85.1(6)
O1B-Pt-C9B	86.6(8)
O3B-Pt-C9B	93.1(7)
O1B-Pt-N1B	89.7(7)
O3B-Pt-N1B	89.8(6)
C8B-Pt-N1B	96.7(7)
C9B-Pt-N1B	173.9(7)
C8B-Pt-C9B	88.6(8)
C8B-Pt-N2B	177.0(8)
C9B-Pt-N2B	94.0(7)
N1B-Pt-N2B	80.8(6)
O2A-O1A-Pt	112.7(10)
O1A-O2A-H0A	109.5
C7A-O3A-Pt	120.0(12)
C4A-N1A-C1A	101.3(16)
C4A-N1A-C3A	107.3(17)
C1A-N1A-C3A	116.1(17)
C4A-N1A-Pt	114.4(13)
C1A-N1A-Pt	103.9(12)
C3A-N1A-Pt	113.5(11)
C5A-N2A-C6A	109.2(17)
C5A-N2A-C2A	118.0(19)
C6A-N2A-C2A	98.8(17)
C5A-N2A-Pt	116.0(15)
C6A-N2A-Pt	108.2(14)
C2A-N2A-Pt	105.0(13)
N1A-C1A-C2A	117.2(18)
N1A-C1A-H1AA	108.0
C2A-C1A-H1AA	108.0
N1A-C1A-H1AB	108.0
C2A-C1A-H1AB	108.0
H1AA-C1A-H1AB	107.2
C1A-C2A-N2A	107.8(17)
C1A-C2A-H2AA	110.1
N2A-C2A-H2AA	110.1
C1A-C2A-H2AB	110.1
N2A-C2A-H2AB	110.1
H2AA-C2A-H2AB	108.5
N1A-C3A-H3AA	109.5
N1A-C3A-H3AB	109.5
H3AA-C3A-H3AB	109.5
N1A-C3A-H3AC	109.5
H3AA-C3A-H3AC	109.5
H3AB-C3A-H3AC	109.5
N1A-C4A-H4AA	109.5
N1A-C4A-H4AB	109.5
H4AA-C4A-H4AB	109.5
N1A-C4A-H4AC	109.5

H4AA-C4A-H4AC	109.5
H4AB-C4A-H4AC	109.5
N2A-C5A-H5AA	109.5
N2A-C5A-H5AB	109.5
H5AA-C5A-H5AB	109.5
N2A-C5A-H5AC	109.5
H5AA-C5A-H5AC	109.5
H5AB-C5A-H5AC	109.5
N2A-C6A-H6AA	109.5
N2A-C6A-H6AB	109.5
H6AA-C6A-H6AB	109.5
N2A-C6A-H6AC	109.5
H6AA-C6A-H6AC	109.5
H6AB-C6A-H6AC	109.5
O3A-C7A-H7AA	109.5
O3A-C7A-H7AB	109.5
H7AA-C7A-H7AB	109.5
O3A-C7A-H7AC	109.5
H7AA-C7A-H7AC	109.5
H7AB-C7A-H7AC	109.5
Pt-C8A-H8AA	109.5
Pt-C8A-H8AB	109.5
H8AA-C8A-H8AB	109.5
Pt-C8A-H8AC	109.5
H8AA-C8A-H8AC	109.5
H8AB-C8A-H8AC	109.5
Pt-C9A-H9AA	109.5
Pt-C9A-H9AB	109.5
H9AA-C9A-H9AB	109.5
Pt-C9A-H9AC	109.5
H9AA-C9A-H9AC	109.5
H9AB-C9A-H9AC	109.5
O2B-O1B-Pt	116.0(11)
O1B-O2B-H0B	109.5
C7B-O3B-Pt	121.9(15)
C3B-N1B-C1B	95(2)
C3B-N1B-C4B	104(2)
C1B-N1B-C4B	120(2)
C3B-N1B-Pt	115.7(14)
C1B-N1B-Pt	108.5(15)
C4B-N1B-Pt	113(2)
C5B-N2B-C2B	111.4(18)
C5B-N2B-C6B	101.8(16)
C2B-N2B-C6B	115.8(18)
C5B-N2B-Pt	117.3(12)
C2B-N2B-Pt	101.0(12)
C6B-N2B-Pt	110.4(13)
C2B-C1B-N1B	120(3)
C2B-C1B-H1BA	107.4
N1B-C1B-H1BA	107.4
C2B-C1B-H1BB	107.4
N1B-C1B-H1BB	107.4
H1BA-C1B-H1BB	107.0
C1B-C2B-N2B	129(2)

C1B-C2B-H2BA	105.2
N2B-C2B-H2BA	105.2
C1B-C2B-H2BB	105.2
N2B-C2B-H2BB	105.2
H2BA-C2B-H2BB	105.9
N1B-C3B-H3BA	109.5
N1B-C3B-H3BB	109.5
H3BA-C3B-H3BB	109.5
N1B-C3B-H3BC	109.5
H3BA-C3B-H3BC	109.5
H3BB-C3B-H3BC	109.5
N1B-C4B-H4BA	109.5
N1B-C4B-H4BB	109.5
H4BA-C4B-H4BB	109.5
N1B-C4B-H4BC	109.5
H4BA-C4B-H4BC	109.5
H4BB-C4B-H4BC	109.5
N2B-C5B-H5BA	109.5
N2B-C5B-H5BB	109.5
H5BA-C5B-H5BB	109.5
N2B-C5B-H5BC	109.5
H5BA-C5B-H5BC	109.5
H5BB-C5B-H5BC	109.5
N2B-C6B-H6BA	109.5
N2B-C6B-H6BB	109.5
H6BA-C6B-H6BB	109.5
N2B-C6B-H6BC	109.5
H6BA-C6B-H6BC	109.5
H6BB-C6B-H6BC	109.5
O3B-C7B-H7BA	109.5
O3B-C7B-H7BB	109.5
H7BA-C7B-H7BB	109.5
O3B-C7B-H7BC	109.5
H7BA-C7B-H7BC	109.5
H7BB-C7B-H7BC	109.5
Pt-C8B-H8BA	109.5
Pt-C8B-H8BB	109.5
H8BA-C8B-H8BB	109.5
Pt-C8B-H8BC	109.5
H8BA-C8B-H8BC	109.5
H8BB-C8B-H8BC	109.5
Pt-C9B-H9BA	109.5
Pt-C9B-H9BB	109.5
H9BA-C9B-H9BB	109.5
Pt-C9B-H9BC	109.5
H9BA-C9B-H9BC	109.5
H9BB-C9B-H9BC	109.5